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SHOCK TUBE STUDIES OF THE IGNITION
OF TRIETHANOL AMMONIUM NITRATE IN NITROUS OXIDE:
PRELIMINARY RESULTS

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JUNE 1990

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U.S. ARMY LABORATORY COMMAND

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This study was initiated following discussions with Nathan Klein of BRL on the decomposition and pathways to ignition of hydroxyl ammonium nitrate (HAN) based liquid propellants. Dr. Klein also provided the samples of triethanol ammonium nitrate (TEAN) crystals. Dr. William R. Anderson of BRL provided assistance with the N_2O decomposition calculations.

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I. INTRODUCTION

Liquid propellants based on aqueous solutions of hydroxyl ammonium nitrate (HAN) and various fuels have been the subject of extensive studies.¹ In particular, the propellants designated LP1845 and LP1846, where the fuel is triethanol ammonium nitrate (TEAN), have been the focus of ignition studies in our laboratory.² It is strongly suggested as a result of these and other studies that the order of involvement of the components is water (evaporation), HAN (thermal decomposition), and TEAN (oxidation/thermal decomposition). Because the TEAN is subjected to the nitrogen oxides formed in HAN decomposition, it is possible that it is either oxidized directly or that it thermally decomposes with its products reacting for energy release. In the present study, we present preliminary results for a study of the direct oxidation of TEAN by the predominant nitrogen oxide expected in this environment, nitrous oxide (N_2O).

II. THE STABILITY OF NITROUS OXIDE

Although one would like to perform these studies in a straightforward experiment where the TEAN is introduced into a flow of the hot oxidizer, some care must be taken. In particular, although N_2O is quite stable under ambient conditions, it is well known to decompose at the temperatures that might be required for rapid ignition of a solid material. For this reason, a brief effort was made to calculate the times over which experiments could be done with hot N_2O before it had substantially decomposed. In addition to the loss of the initial oxidizer, one must also be concerned with the growth of product species which might be much more reactive. Although any decomposition of the oxidizer is of course a part of the reaction process, it is desirable that any observations are made with a knowledge of the composition of the gases present.

Calculations to model the thermal decomposition of N_2O were performed using the CHEMKIN package of subroutines.³ A simple model was used by adapting a sample problem at the end of the manual. We assumed a zero dimensional, homogeneous distribution of all reactants under constant pressure, adiabatic conditions. The kinetic scheme and associated rate constants which were used are given in Table 1. Values were taken from the literature, but this was not intended to be a rigorous modeling effort; rather it was simply desired to get an order of magnitude estimate of the time available for observations in hot N_2O .

Typical results for the mixtures and temperatures of these experiments are shown in Figure 1. Two major trends are quite apparent in this figure. The first is that with increasing temperature, the amount of time available for experiment before major loss of N_2O goes down rapidly. The second trend is that available time goes down rapidly with pressure. Not shown here is the additional dependence of N_2O lifetime on dilution. As the N_2O is diluted further by argon, the lifetime also increases. In all cases, it is quite clear that there is probably not time available for a heated flow experiment. In fact, following these calculations, it was decided that this study could probably be performed best in a shock tube where the temperature and pressure are raised almost instantaneously on the time scales of expected reactions. As a shock tube was becoming available for ignition studies at the time of these calculations, studies were pursued with it.

Table 1. Kinetic Pathways and Rate Parameters for N₂O Decomposition Modeling

Reaction	A Factor	b	Ea
$N + O_2 = NO + O$	6.40E09	1.0	6280.
$N + NO = N_2 + O$	3.30E12	0.30	0.
$NO_2 + M = NO + O + M$	1.10E16	0.	66000.
$NO_2 + O = NO + O_2$	1.00E13	0.	600.
$N_2O + M = N_2 + O + M$	1.60E14	0.	51600.
$N_2O + O = NO + NO$	1.00E14	0.	28200.
$N_2O + O = N_2 + O_2$	1.00E14	0.	28200.

III. EXPERIMENTAL

The shock tube used in these studies is stainless steel with a 98 mm inside diameter. The driver section is 1.2 m long; the driven (test) section is 6.2 m long in the configuration used in the present study. For "typical" test conditions, with a 0.0075 inch thick Mylar polyester diaphragm, an initial test section pressure of 70 Torr (9.2 KPa) and a driver pressure of 70 psi (470 KPa) of helium yield an incident shock Mach number near 2.9. Calculated temperature and pressure values behind this shock are 804 K and 0.93 atm (93 KPa). The calculated values after passage of the reflected shock for these same initial conditions are 1360 K and 4.5 atm (450 KPa). Pressure gauge measurements have been used to verify that the actual values reached are near the values calculated over the range of interest here. Various combinations of initial test pressure and diaphragm thickness are used to obtain desired pressure/temperature combinations. Using pure helium as the driver gas gives useful test times on the order of one millisecond; these values are adequate for ignition times here, which are typically 500μs or less.

The layout of the test end of the shock tube is shown in Figure 2. The main instrumentation consists of piezoelectric pressure transducers mounted at positions 55 and 768 mm from the end wall to follow the shock wave propagation and a 1P28 photomultiplier tube (PMT) which is mounted to record the light through a window in the end wall of the shock tube. Light detected by the PMT is filtered by neutral density filters (typical density 2.0 to 3.0) and a red-pass colored glass filter which passes light below 550 nm. It is expected that the detected light during combustion is dominated by sodium emission. The output of the pressure transducers and PMT are recorded by a digital scope; the records are ported to a computer for analysis as required. The sample of TEAN to be ignited is placed on a one inch diameter disk with feathered edges mounted in the center of the tube 210 mm from the end wall. The TEAN is ground to a fine powder of unknown particle size; typical sample size is about 20 mg. In the course of an experiment, the incident shock sweeps the powder off the disk and disperses it in the region between the disk and the end wall. The experiment may be done such that the incident pressure and temperature are high enough to ignite the sample. Alternatively, and in most of the present observations, the incident temperature rise can be low enough that the reflected shock and its associated temperature rise are

required to ignite the sample. In a typical case near 1360 K, TEAN particles moving with the gas velocity behind the incident shock interact with the reflected shock wave approximately 500 μ sec after passage of the incident shock. Pressure records with the disk present do not differ measurably from those without, indicating that perturbation to the flow is minimal. Thermochemical calculations are made of the characteristics of the incident and reflect shock to obtain predicted pressure and temperature conditions in the tube. The pressure is measured directly; as long as these measurements agree with the calculations, it is assumed that behavior is reasonably ideal and that the predicted temperature is achieved.

IV. OBSERVATIONS

A typical photomultiplier record is shown in Figure 3 for a calculated⁴ reflected shock pressure of 4.6 atmospheres (0.46 MPa) and a temperature of 1460 K. Zero time in this and other similar plots is the calculated time that the smallest TEAN particles, those assumed to move with the gas velocity behind the incident shock, interact with the reflected shock. The two curves are clearly distinguishable by intensity; however, a careful comparison of the intensities and shapes at early times shows little difference in that region. Attempts to subtract the background from the signal has shown that very small changes in the background make big differences in the apparent time of onset of ignition based on the light signal. Thus, unless either the background light can be greatly suppressed or much better reproducibility can be achieved, quantifying measurements such as this will be difficult. Ignition at a slightly lower temperature and pressure is shown in Figure 4, with values of 3.2 atm (0.32 MPa) and 1460 K. In this second case, the delay before ignition is sufficient to separate the resulting light from the background. Also shown in the figure is the difference between the ignition signal and the background. For these conditions, this difference curve, including the negative portion, has been found to be quite reproducible. The negative portion of the difference curve is probably due to a decrease in the background when the TEAN is present. The cause is unknown; it may be due either to cooling or obscuration. In cases such as this one, it is clear that one could define a consistent and reproducible criterion for ignition.

In addition to the evidence of ignition from the emitted light, pressure records show clear increase in pressure when ignition occurs, especially when it occurs past the peak pressure. In Figure 5 is shown the pressure differential from an event at 1350 K along with the corresponding light emission. As can be seen, the pressure pulse at station one and light emission are well correlated. In general such pressure differences show this behavior in these experiments; however, the signal to noise can be much less than desired. In the example shown in Figure 5, the additional pressure from TEAN combustion is near one atmosphere, almost one-fourth the maximum pressure of the reflected shock.

A limited number of observations were made with a similar mixture of oxygen in argon, the goal being to compare light and pressure signals if ignition were to occur. Based on light emission records, the main observations were that (1) there is ignition in oxygen with corresponding light intensity well above the background level and (2) the ignition takes place at lower temperatures and probably with shorter delay times than with N₂O under the same conditions.

V. DISCUSSION

While it is clear from both the light emission and pressure records that ignition of the TEAN is occurring, some ambiguity of the significance of these observations remains. The first is related to the degree and effect of any decomposition of the nitrous oxide. Clearly further studies in the characterization of the gas composition versus time needs to be done. This goal probably can be met by diagnostics on the presence of NO_2 or other products in the decomposition process. Refinement of the model rate constants and pathways of decomposition could also be pursued to determine probable limits of composition versus time. The observation that oxygen ignition is more prompt than with N_2O suggests that the N_2O decomposition either is not as fast as we calculate or the products do not substantially accelerate the ignition reactions. This question can also be addressed by decreasing the concentration of oxidizer in the inert gas.

Another element of this work which requires refinement is the definition of time to ignition of the solid material. The signal-to-background ratio may be greatly improved by use of a narrow band optical filter at the wavelength of some key combustion radical such as OH or CN. In this case one could use the earliest light as evidence for ignition of the smallest solid particles. Alternative definitions are being developed. This definition is critical to establishment of ignition delay versus temperature and pressure. The use of high-speed photography for distinction by spatial location of the light emission is also a possibility.

A third question which remains to be addressed is whether the TEAN is reacting as a solid or first undergoes thermal decomposition followed by gas phase reactions. Attempts will be made in continuing studies to characterize the process more fully, as well as to measure the kinetics parameters.

VI. SUMMARY

We have shown that TEAN powder ignites and burns with significant light emission and pressure generation in a nitrous oxide/argon mix. Ignition threshold appears to be above 1300 K. Ignition delay is a function of the temperature and pressure of the gas. Ignition in oxygen takes place in shorter times and at lower temperatures than with N_2O . Our calculations indicate that the thermal decomposition of the N_2O may be important in our observations. Studies are presently underway to address various aspects of these observations.

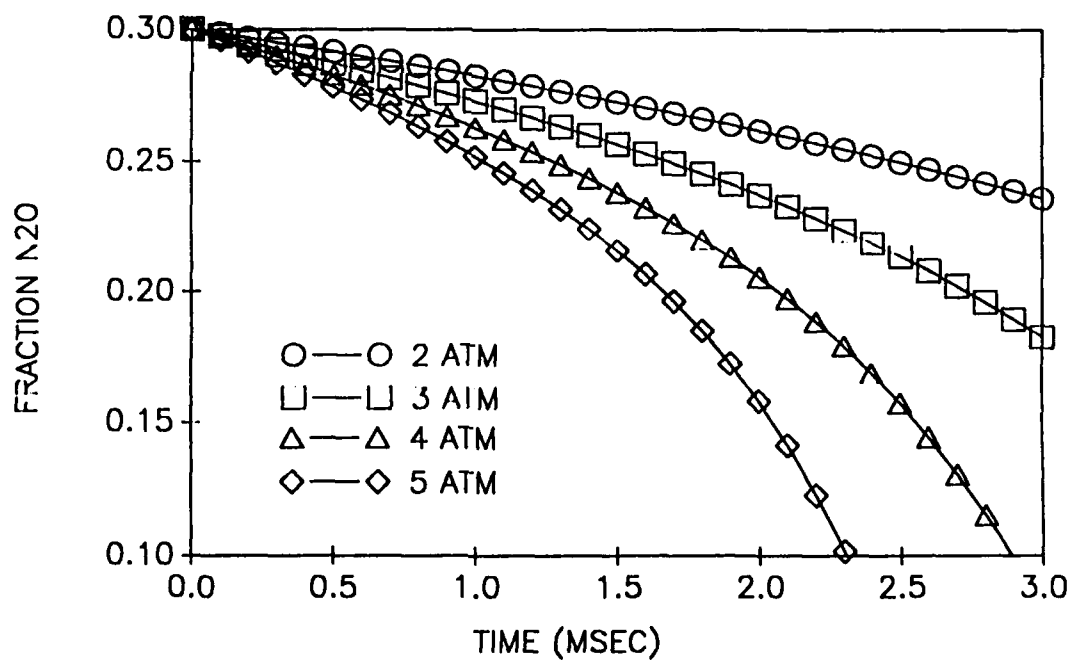
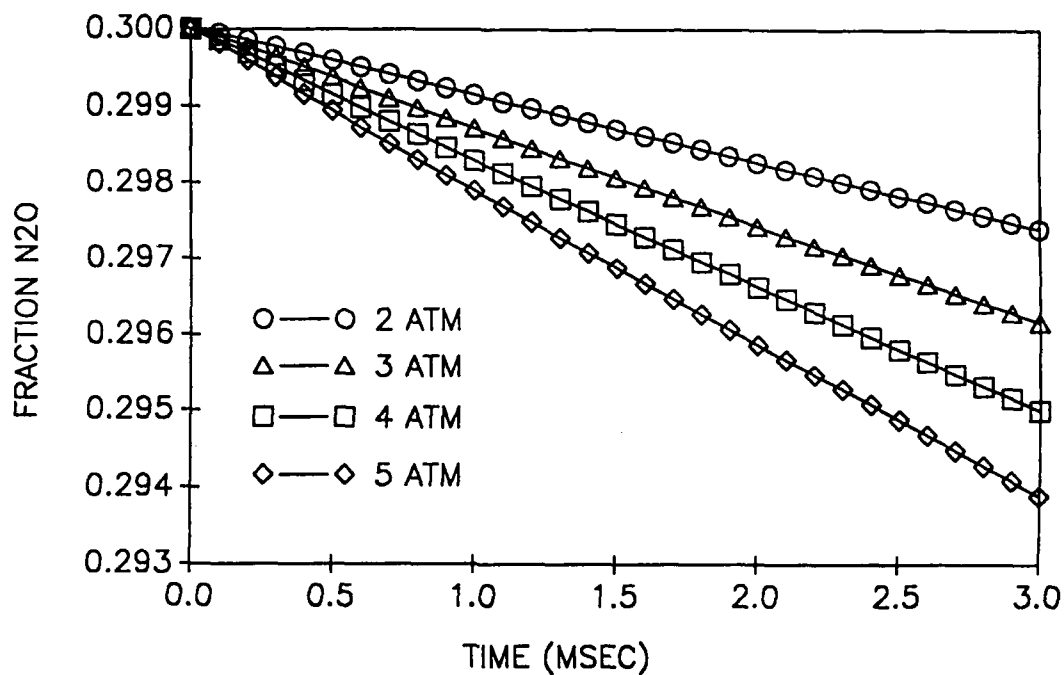


Figure 1. Calculated fraction of N_2O as a Function of Time for Various Pressures at (a) 1200 K and (b) 1400 K

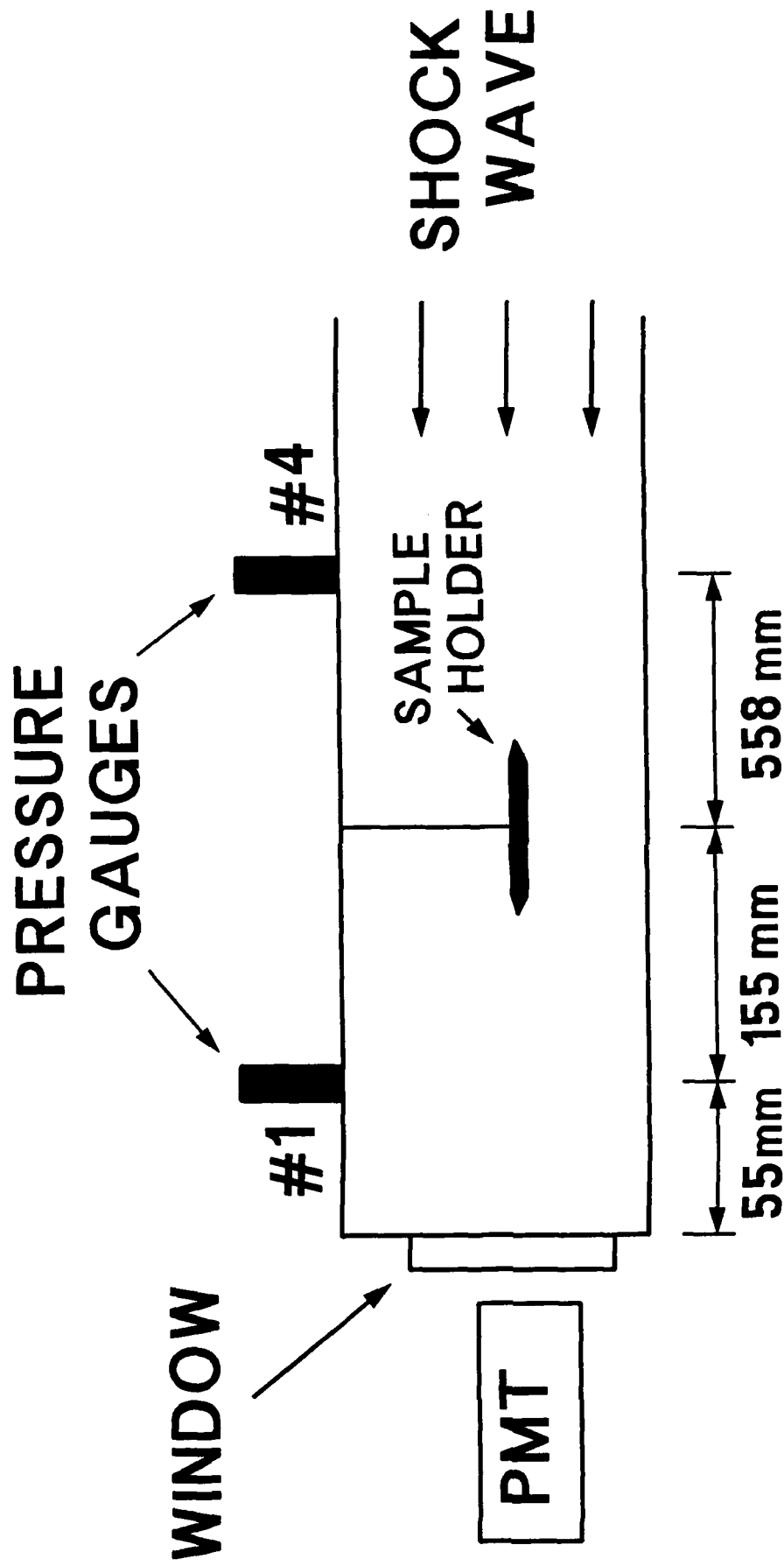


Figure 2. Schematic Diagram of Test End of Shock Tube

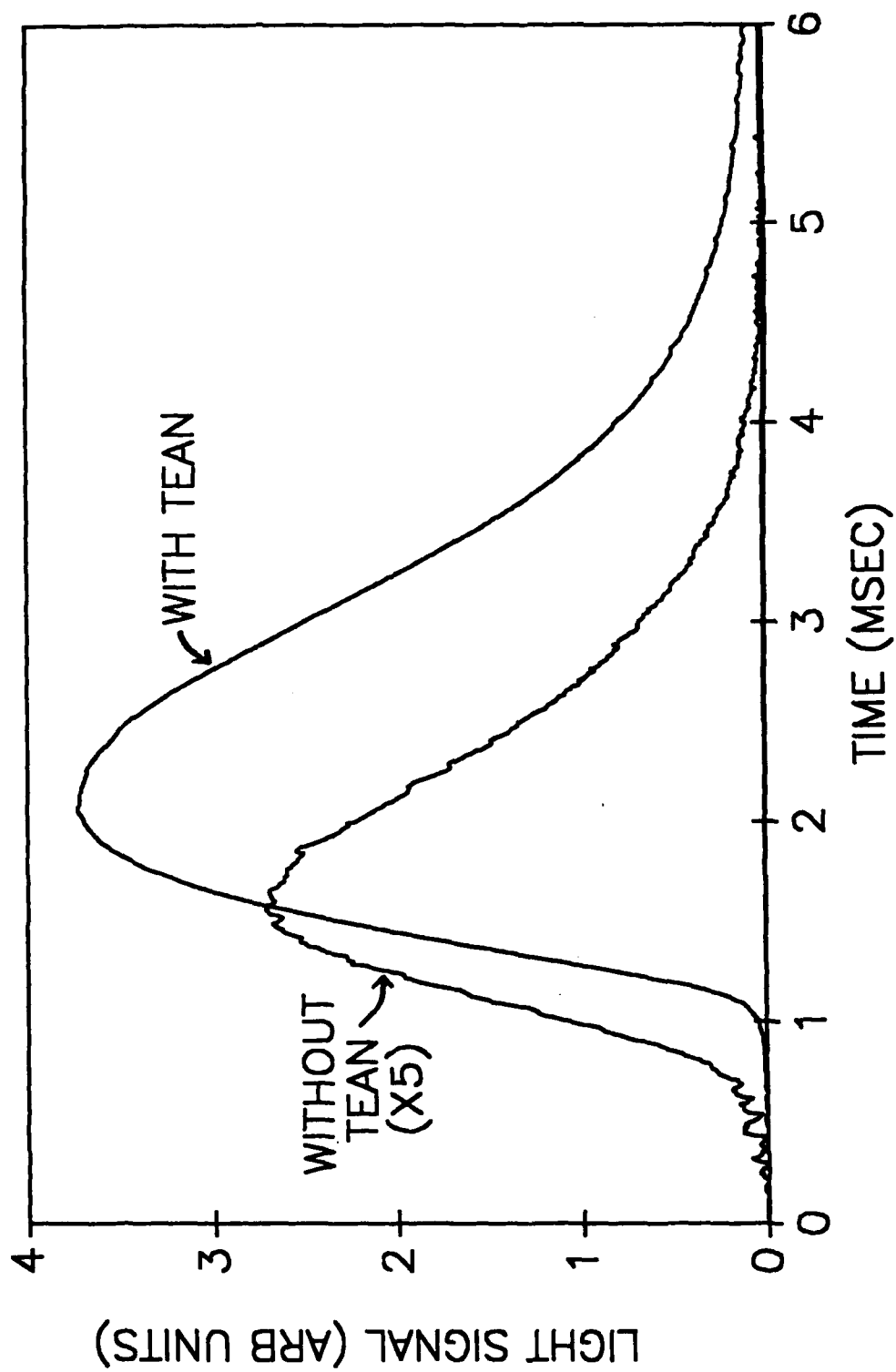


Figure 3. Light Signals With and Without TEAN in N_2O Shocked to 1500 K and 4.6 Atm Pressure

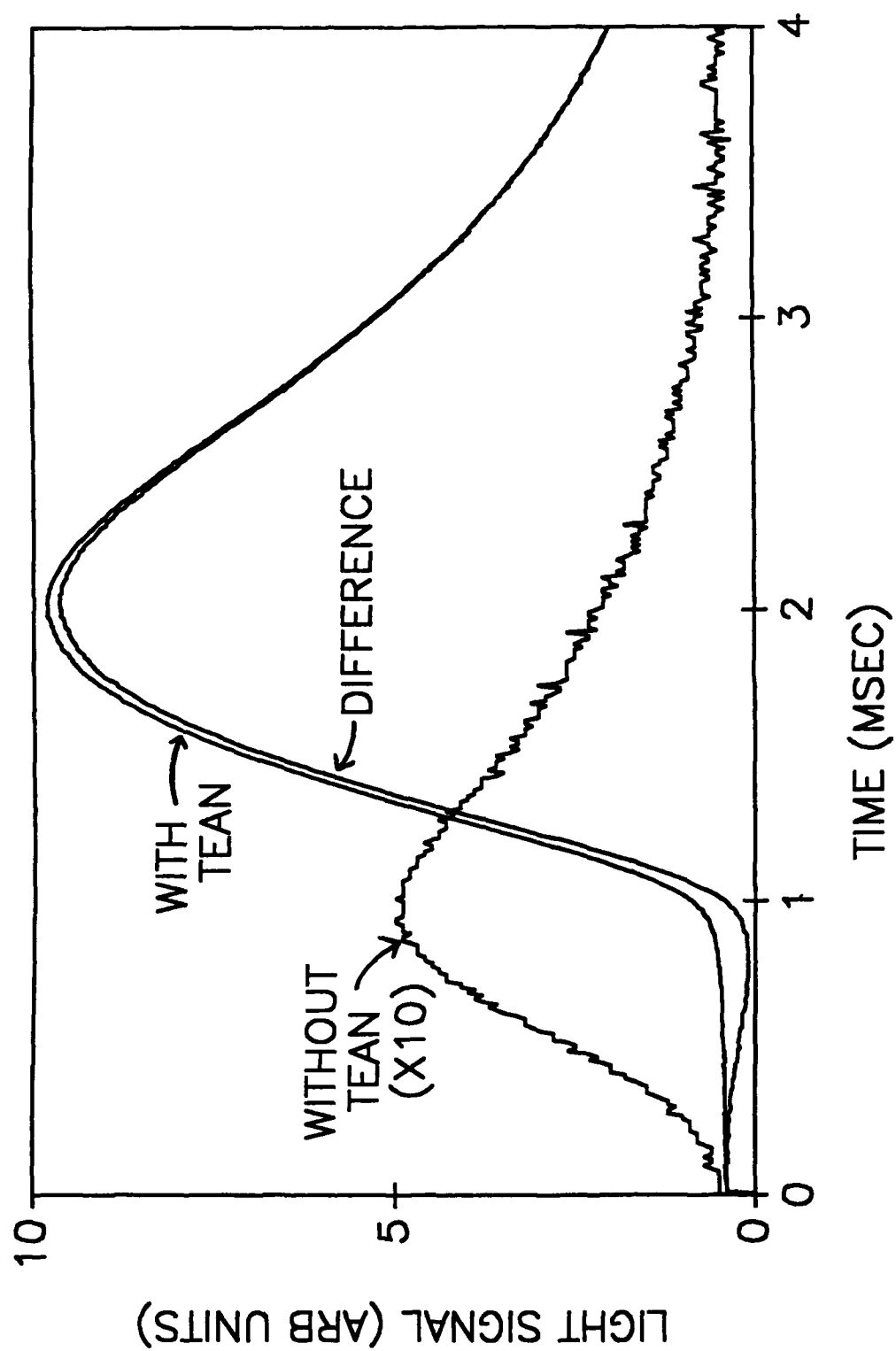


Figure 4. Light Signals With and Without TEAN in N_2O Shocked to 1460 K and 3.2 Atm Pressure

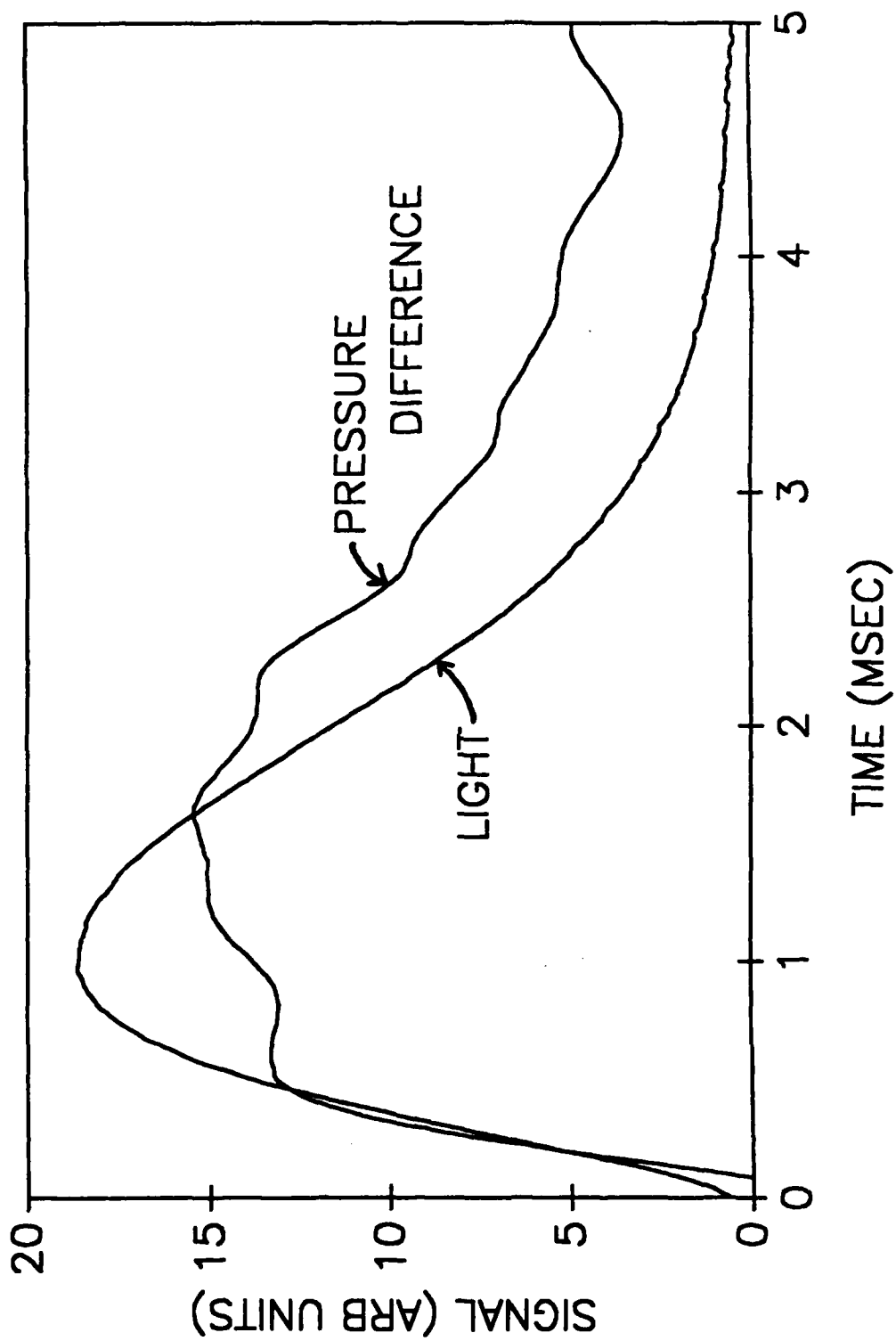


Figure 5. Difference of Pressure Pulses and Light Emission from TEAN in N_2O Shocked to 1350 K and 4.5 Atm

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